Polypropylene composites obtained from self-reinforced hybrid fiber system

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ABSTRACT: The purpose of the study was to obtain a composite material with the self-reinforced structure, which processing provide increased mechanical properties. The composites used in presented work were prepared from the two types of fiber mixtures, both were based on polypropylene fibers, the difference was in used cellulose or wood flour filler. Composites were prepared using the hot compaction method. The presented research describes the effect of the composite composition and processing conditions. The results include the static tension measurements, tensile impact tests and thermal analysis, including: DSC and DMTA. The structure has been studies using the SEM observations. Results of presented studies confirm the self-reinforcing effect in obtained hybrid composites. It provides in the comparison to the standard wood polymer composites to the higher level of material reinforcement with lower amount of natural filler. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43283.

INTRODUCTION

In the last 20 years, there has been tremendous interest in the use of natural fibers for the manufacturing of polymer composites.[1–6] The hybrid composites based on polymer matrix and natural fibers such as wood flour and cellulose flour are valued materials with a great mechanical and thermal properties as well as enormous prospective applications in many areas of life.[7–11]

Polypropylene is a thermoplastic polymer being widely applied in the industry and is usually combined with inorganic particles or fibers to improve its mechanical properties and reduce cost. This material may occur in various physicochemical forms.[12,13] The bicomponent fibers of polypropylene are characterized by unique structure.[14–16] The complexity of this material allows to obtaining self-reinforced composites with special mechanical properties. The structure of bi-component fibers is based on a combination within one filament of two varieties of the same polymer. The core fibers are often the homopolymer, the shell/sheath is formed by a low melting copolymer. Such a construction of the fiber does not lower its strength values while facilitating processes of bonding woven and non-woven fabrics made of bicomponent fibers.

The mostly used inorganic fillers group include calcium carbonate,[17] mica,[18] or talc.[19] The replacing the inorganic fillers by

lignocellulosic materials would be highly beneficial from the point of the environmental sustainability.[20,21] The biodegradability of the natural fibers is considered as the most important and interesting aspects of their utilization in polymeric materials. However, there exist many others advantages, such as easy availability, low cost, reduced tool wear, and acceptable specific strength, along with their renewable and degradable features are some of the important properties of the natural fibers, which make them suitable to use as filler/reinforcement materials in polymer composites.

The processing conditions play, next to the mechanical properties of natural fibers, an important role for industrial use of these materials.[22] The hybrid composites can be processed with the already commonly applied methods: compression moulding,[23–25] pultrusion,[4,26,27] extrusion,[28] filament winding, and others. In order to obtain the hybrid polymer composites, in this research the compression molding method was used. The main reason for this choice was the precision of processing conditions during the material compaction. In addition, the objective of this study was to investigate the possibility of using this type of production process as a method of polymer composite preparation.

The topic of the hybrid composite systems has been known for at least two decades. The development of research directions of
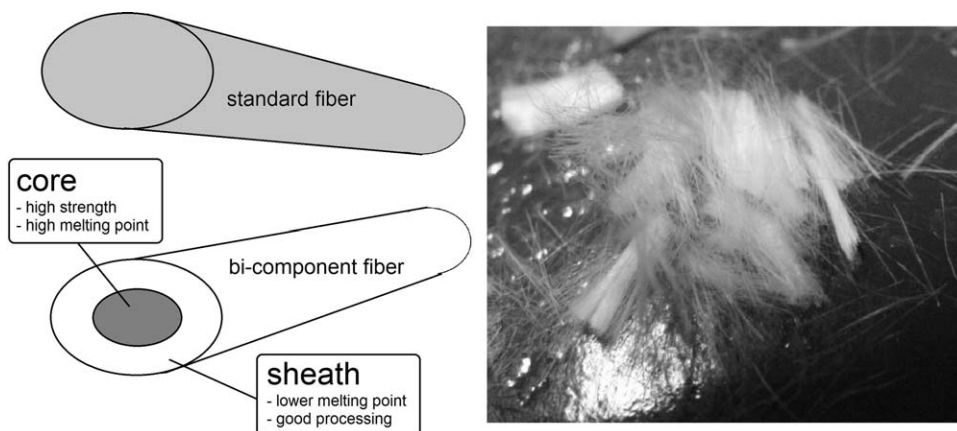
Figure 1. The structure of the bicomponent fibers and macroscopic view at the input material.

such a materials has led to a wide spread of this issue and resulted in taking this terminology of new materials, far from primary issue. A literature review indicates that the original use of the term "hybrid composite" in the context of reinforced polymer composites, where the use of several types of fillers favorably modified the properties of the materials, as an example can be listed here the subject of use of the natural and glass fiber mixture reinforcing the thermoplastic polymers.[29] This issue has expanded over time as technology development has allowed the use of nanofillers.[30] In the last few years the subject of hybrid composites spread on classical fillers such as chalk or talc surface-modified by organometallic compounds,[8,31,32] a separate group are also functional silane compounds modified during the stage of synthesis.

In the present study, the issue of hybrid composites is described in the original sense, including the use of different types of reinforcing fibers in order to achieve improved properties of the resulting product. Basic and key materials in the presented studies are bicomponent fiber. The use of this specific material has two main functions. The first one resulting from the oriented fiber structure is a function of the reinforcement. The second one resulting from the application of two-component structure is the formation of composite matrix.

The concept of presented hybrid composite and the preparation using the hot compaction method is approaching the tested materials to the self-reinforced composites.[33,34] The use of two component materials for the preparation of self-reinforced composites has been described for the materials obtained by compression moulding,[15] injection molding,[31,35–37,39] or even selective dissolution.[40] In this paper the results of the cited works are simultaneously confronted with the idea of hybrid strengthening. In the presented studies, bicomponent fibers serve as a source of matrix for natural wood fibers and pure cellulose.

The main aim of this work will be to obtained the self-reinforced hybrid composites based on polypropylene matrix and natural fibers (wood flour and cellulose flour) using the compression molding method. The obtained results will be helpful to evaluate the usefulness of the tested materials and proposed manufacturing method.

EXPERIMENTAL PROCEDURE

In this study, the influence of the filler type and content as well as different conditions of processing on mechanical and thermal properties of obtained hybrid composites will be determined. The values obtained can be used to estimate yield stress, E modulus, and impact strength of produced composites.

Materials

Bicomponent Fibers. Polypropylene based bicomponent fibers were used as a main component for all prepared composites. Fibers were provided by the company MiniFibers. The fiber characteristic include: diameter = 16 μm, length = 5 mm, tensile strength = 190 MPa. The declared melting point was 165°C for the core and 135°C for the sheath. The volume proportion of core/sheath was 50%/50%. Fibers structure and appearance are presented in the Figure 1.

Natural Fillers. Lignocel C-120 natural wood fiber material was used as a filler to obtain hybrid composite systems. It is produced from untreated solid wood obtained from the conifer trees of few species. Lignocel C-120 was obtained from J. Rettenmaier and Söhne GMBH+CO.KG. This compound with characteristic odor is insoluble in water and characterized by light yellow color. Lignocel C-120 is highly absorbent and dust free.

Arbocel FD 600/30 natural cellulose fibers served a function of filler in hybrid composites. It is a pure substance gained from renewable raw materials and produced by J. Rettenmaier and Söhne GMBH+CO.KG. Arbocel is a white and odorless powdery material. In this form Arbocel cellulose is slightly hygroscopic, its moisture content is approximately 10–12%. In addition, this material is insoluble in water and organic solvent. This substance is characterized by resistant to dilute acids and bases. The main physical and chemical properties of the Arbocel and Lignocel are collected in the Table I.

Composite Preparation

The fabrication of specimens consisted of a two-step process of preparing the hybrid composite. The preliminary stage included the preparation of the material by mixing in a high speed blender at 3500 rpm for 2 min and drying at 60°C for 10 h in cabinet dryer, which would prevent the formation of pores during the compaction of the material. After drying, the mixture was

Table I. Basic Physicochemical Parameters of Natural Fibers

	Wood flour—lignocel	Cellulose flour—arbocel
Particle size	70-150 µm	50-100 µm
Bulk density	110-145 g/l	110-145 g/l
Humidity	6%	10%
pH (20°C)	4.5-6.5	5-7

weighed in amount about 20 g for each sample. The final stage of composite shaping was preceded by preheating the mold of hydraulic press to the temperatures of bonding process. Then, the material was placed between two heated plates of compression molding press at 155, 165 and 175°C respectively, for 10 min with the pressure of 7 MPa acting on the material. The cooling stage took place at the same pressure for 20 min. The dimension of wood flour/cPP-PP and cellulose flour/cPP-PP composite samples was 100 × 100 mm and 1 mm thickness. After that all the composites were cut into required dimensions for further tests. A detailed list of the obtained samples with their designations and the preparation conditions are summarized in Table II.

Materials Characterization

DSC. In this research, Netzch Phoenix F1 DSC 204 was used to analyze the thermal properties. The temperature program was

exactly the same for all samples. The temperature range of measurements was between 20 and 230°C, at heating/cooling rate of 10°C/min. The average weight for each sample was 5 mg. The measurements were conducted in the aluminum pans under the inert atmosphere of nitrogen.

DMTA. The dynamic thermomechanical analysis was carried out using Anton Paar MCR 301 instrument. The analyses were performed at a frequency of 1 Hz with a heating rate of 2°C/min over temperatures from −50 to 160°C. Samples with dimensions of 30 × 8 × 1 mm were tested using a torsion clamp system.

Mechanical Tests. Tensile tests were performed using an Instron 4481 universal instrument (load cell = 50 kN), in accordance with ISO 527-1 standards, dumbbell sample (type 1BA, gauge length = 25 mm) were cut from the obtained plates. The test speed was 10 mm/min. The stress–strain curves were obtained and the modulus and yield stress values were evaluated. Minimum 10 samples were used for each test series.

Impact tests were performed using pendulum testing hammer (model PW 5) in accordance with ISO 8256 standards. The tensile-impact strength defined as the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. The use of this method was dictated by the difficulties

Table II. Composition and Processing Conditions Used on the Preparation of the Lignocellulosic Flour/Polymer Composites

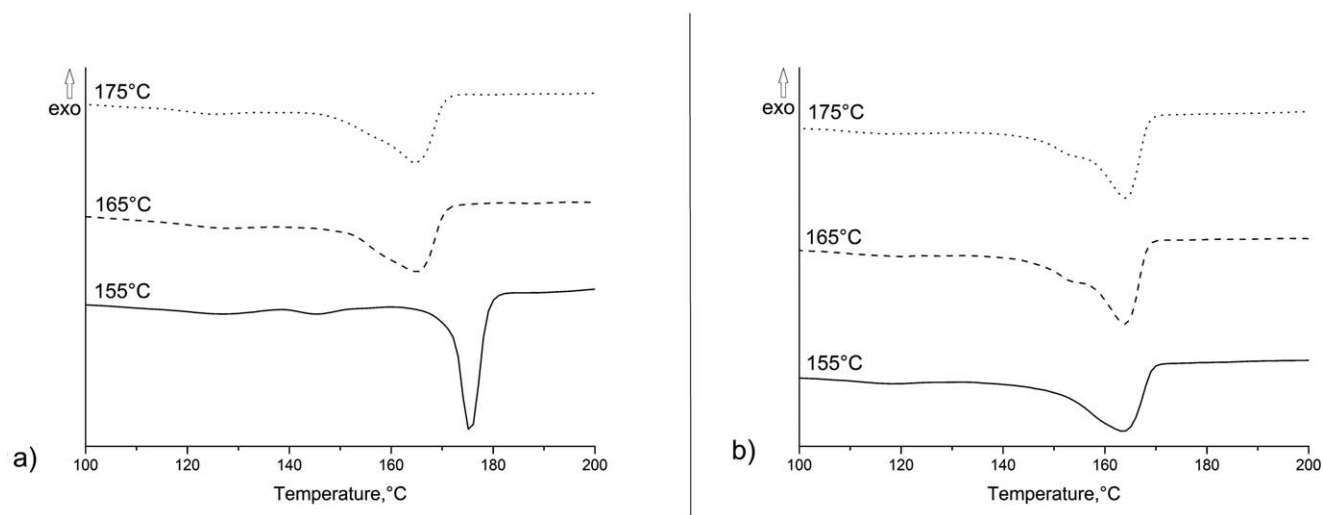| Material	Bicomponent fibers content (%)	Wood flour content (%)	Cellulose flour content (%)	Compaction temperature (°C)
Pure bicomponent fibers				
cPP/PP(155)	100	–	–	155
cPP/PP(165)	100	–	–	165
cPP/PP(175)	100	–	–	175
Wood flour filler				
WF10(155)	90	10	–	155
WF10(165)	90	10	–	165
WF10(175)	90	10	–	175
WF30(155)	70	30	–	155
WF30(165)	70	30	–	165
WF30(175)	70	30	–	175
WF50(155)	50	50	–	155
WF50(165)	50	50	–	165
WF50(175)	50	50	–	175
Cellulose flour filler				
CF10(155)	90	–	10	155
CF10(165)	90	–	10	165
CF10(175)	90	–	10	175
CF30(155)	70	–	30	155
CF30(165)	70	–	30	165
CF30(175)	70	–	30	175
CF50(155)	50	–	50	155
CF50(165)	50	–	50	165
CF50(175)	50	–	50	175

Figure 2. The heating thermograms for composites obtained form cPP/PP fibers at different temperatures, thermograms from (a) the 1st heating stage and (b) 2nd heating stage.

using the Charpy hammer. The tensile-impact strength method was more suitable for 1 mm sample thickness.

SEM Observations. The microstructural analysis of the fracture surface of the composites was carried out by means of a scanning electron microscope—Carl Zeiss EVO 40. The samples were initially freeze-fractured under liquid nitrogen and then the fractured surface was coated with a fine layer of gold before observation. The morphology and microstructure composites have been studied in cooperation with the Collegium Biologicum (Adam Mickiewicz University).

RESULTS AND DISCUSSION

Thermal Properties

In this research, thermal analysis method DSC was used to explain the course of the thermal properties changes of the analyzed material. From the observations of samples with different ratio of matrix and filler, very similar DSC thermograms for all composite samples were noted. It can be explained by inert nature of wood and cellulose fibers on the DSC signal. The visible peaks of melting and crystallization relate to bi-components fibers made from polypropylene. Therefore, in the presented results of DSC was shown only main, repeated trends in composites samples. In the Figure 2 were shown the DSC thermograms of composite samples consisted of cPP/PP fibers, without additives.

The DSC results derived from 1st heating cycle [Figure 2(a)] give a clear difference in the obtained curve for compressed samples at 155°C. The maximum of melting point was shifted by around 10°C, above the maximum of temperature for the other two samples. In comparison with compressed samples at 165 and 175°C which show typical curves for polypropylene fibers where the maximum of peak was observed at around 165°C with very similar distribution of melting enthalpy.

On the other hand, the DSC results from 2nd heating cycle [Figure 2(b)] show melting curves having identical shape for all

tested samples. This is a confirmation of direct influence of thermal conditions on properties of oriented fibers.

The essential feature of processing is temperature control which ensure a high level of fiber orientation. In the case of cPP/PP(155) samples a different shape of DSC curve was observed, in comparison with samples other two composites, cPP/PP(165) and cPP/PP(175). It can be explained by the presence of oriented polypropylene fibers prevented from the relaxation by lower temperature of processing. During the measurement the stress accumulated in the fibers caused a shift of temperature peak.

Figure 3 shows the DSC course of selected samples filled with wood flour and compressed at 155°C. The pure cPP/PP composite curve is used as a reference and placed at the top of the thermograms chart. Despite of identical thermal conditions, the melting peak is different for composite material. What more, the highest melting temperature was obtained for samples
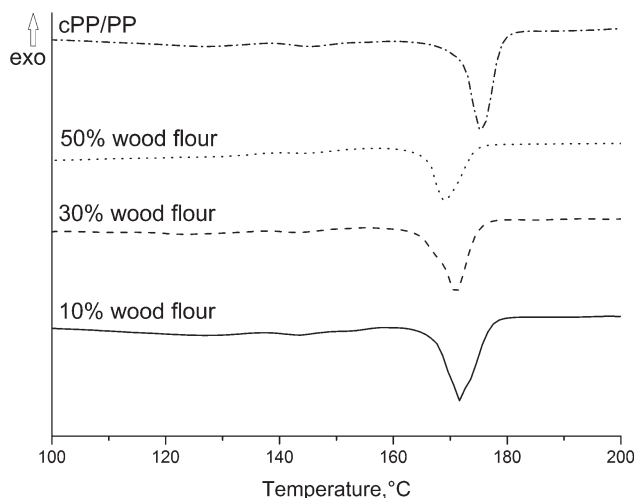

Figure 3. The comparison of the 1st heating thermograms of the wood flour filled composites, compacted at 155°C.
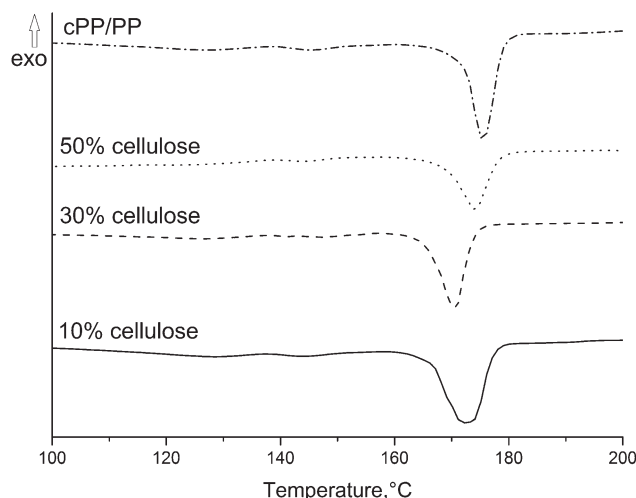
Figure 4. The comparison of the 1st heating thermograms of the cellulose flour filled composites, compacted at 155°C.

without filler and the lowest melting temperature was achieved for composites filled with wood flour of 50 wt %. It can be observed a strong trend of the decreasing melting temperature with increasing filler content. There was also observed decrease of melting enthalpy that is associated with reducing amount of matrix in the composites.

In the case of DSC thermograms for composites filled with pure cellulose (Figure 4), as previously mentioned—the highest melting temperature was obtained for samples without filler. This time, cannot be observed any clear trend for samples with different amount of filler. However, the common trend for all prepared composites is a constant shift of melting temperature above 170°C in the presence of oriented fiber structure.

Thermomechanical Analysis

The comparative analysis of storage modulus and tan δ (Figure 5) refers to prepared cPP/PP samples at various thermal conditions of compression molding process. The expected differences

in the DMTA thermograms had indicate the trend of mechanical properties associated with the changes in structure of the material as a function of processing temperature.

The results of the DMTA analysis for the hybrid composites material filled with wood flour are plotted as dependency of storage modulus G' or tan δ at a reference temperature of 155 and 175°C (Figure 6). In order to have sufficiently clear presentation of results, samples with 10 and 50 wt % content of fiber were placed on the graph. The obtained thermograms were compared with reference samples of the neat cPP/PP fibers.

The comparison of storage modulus (G') indicates the effects of the additive of wood particles on stiffness of hybrid composites [Figure 6(a,b)]. For compressed samples at temperature 175°C [Figure 6(b)], the values of storage modulus clearly increases across the whole measuring range which is related with increasing level of wood flour in the polymer composites.

The situation is slightly different for composite compacted at 155°C [Figure 6(a)]. The observed differences are clearly for the benefit of hybrid composites. On the other hand, the differences between materials with 10 and 50 wt % of wood were observed only above the glass transition temperature. At the glassy state, the storage modulus curves of both composites are similar, after reaching the T_g at around 0°C, curves of G' behave in a very predictable way and refer to samples received at 175°C.

The diagrams shown in Figure 6(c,d) represent mechanical loss factor (tan δ) and confirm a little different nature of changes in mechanical properties of composites samples which were obtained at various conditions of compression molding. While the charts representing the compression temperature of 175°C [Figure 6(d)] are evidently showing the reinforcement of materials with increasing amount of wood flour. Additionally, the field of area under maximum peak of T_g decreases with growth of filler concentration. On the other hand, curves representing the samples compressed at 155°C [Figure 6(c)]; it means cPP/PP(155) and WF10(155) have a similar shape of thermogram curves, which would indicate an approximate effect of
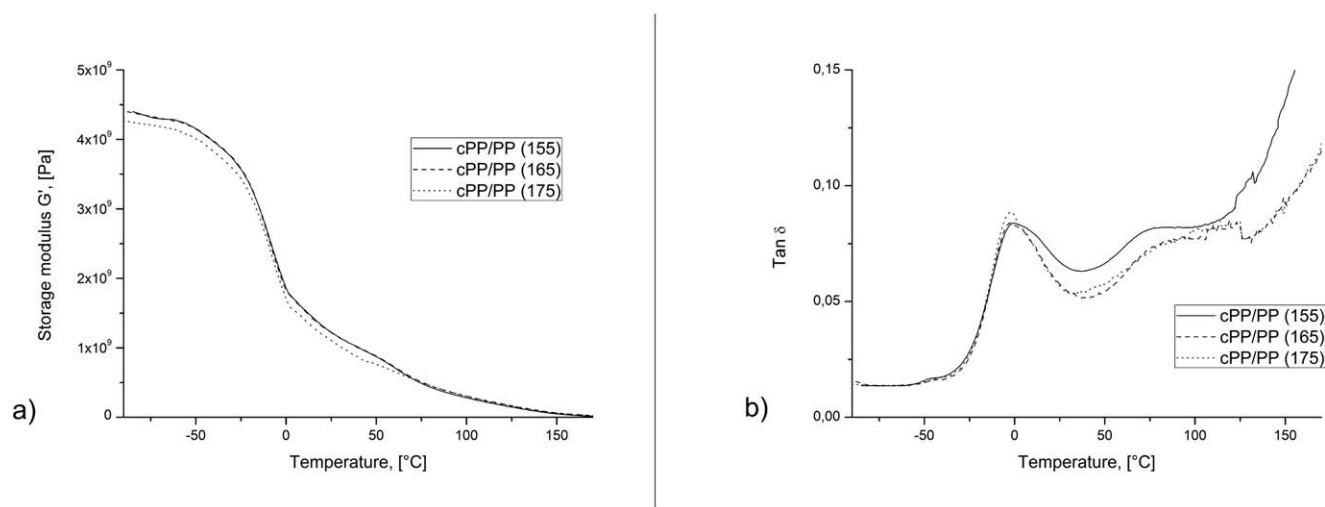

Figure 5. The DMTA analysis of the composites obtained from cPP/PP fibers: (a) storage modulus curves and (b) tan δ curves, thermograms are related with different compaction temperatures.
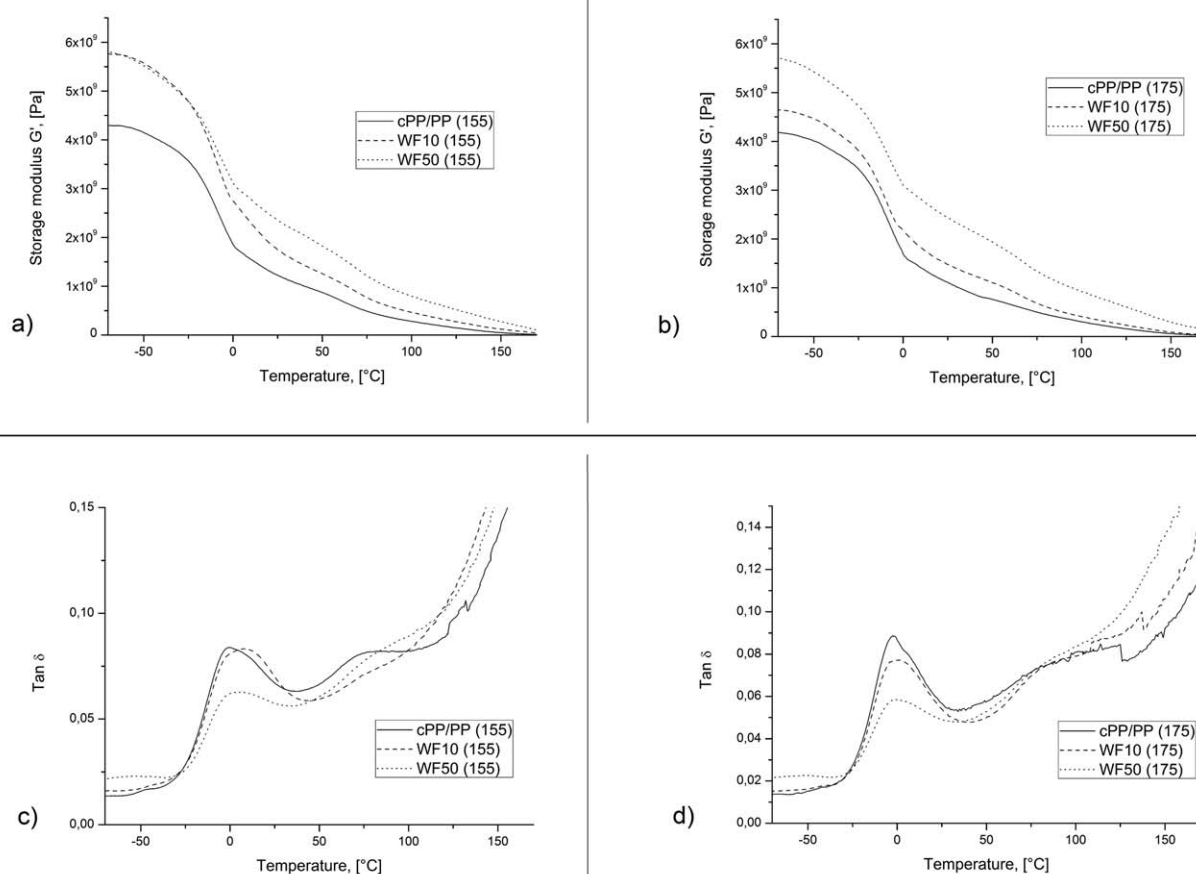
Figure 6. The comparison of the storage modulus (a,b) and tan δ (c,d), the charts differs the processing temperature 155°C for a,c and 175°C for b,d.

reinforcement for wood and polypropylene fibers. The increase of wood content to 50 wt % made the composites much more strengthened—it can be observed by reduction of area under the curve.

Since the measurements for composites sample filled with pure cellulose were achieved using the same conditions just as the samples filled with wood flour, the analysis of DMTA refers primarily to the composite with wood fibers. The results analysis for the cellulose filled composites revealed the similar trends as for the presented wood flour based materials.

Mechanical Properties
The results of mechanical tests were summarized and presented in Table III. The selected values from the tables are presented in the form of graphs in the figures below.

Figure 7(a,b) is representing the yield stress and E modulus of the wood flour filled composites. It is worth to mention that significant increase of yield stress affects samples containing 10 wt % of the wood fibers in the composites. This growth was achieved for compressed samples at 155 and 165°C. In these conditions of preparing composites, a small addition of wood particles to polymer composites leads to double increase of the yield stress. Nevertheless, the plot of received samples at the highest temperature of processing (175°C) indicates a trend of

decreasing of yielding stress with increasing amount of filler. In regard to previously samples, these detentions were observed at higher degree of filling. Finally, the composites with 50 wt % content of filler showed reduced values of yield stress, because of poor interfacial adhesion at the interface between polymer and wood.

In connection with measurements of E modulus [Figure 7(b)] the values slightly decreases in compare with unfilled samples which were obtained at analogues conditions, for the highest temperature of processing was observed a gradually increase of E modulus with rising content of wood fibers.

The measurements of yield stress for composites filled with cellulose present similar trends [Figure 7(d)]. In the case of yield stress analysis concerning hot compacted sample at 155°C was observed a significant increase of strength for 10 wt % cellulose content. For the other samples compressed at temperatures 165 and 175°C is maintained a continuous decrease of strength caused by the increase of filler content. The analysis of modulus changes [Figure 7(e)] do not show clearly answers about the relationship relating to the polymer composites filled with pure cellulose. In addition to the general dependency between increasing modulus and growing amount of filler, other changes base on the E modulus values do not introduce any trends.

Table III. Static Tension and Tensile Impact Tests Results of Prepared Composites Samples

Material	Yield stress (MPa)	E modulus (MPa)	Strain at peak (%)	Strain at break (%)	Impact strength (J/mm^2)
		Hot compaction at 155°C			
cPP/PP(155)	24.1 ± 0.5	1052 ± 100	12.5 ± 6.5	21.5 ± 5.5	0.69 ± 0.07
WF10(155)	42.8 ± 3	990 ± 78	28.5 ± 2	29.5 ± 2.5	0.61 ± 0.06
WF30(155)	26 ± 4.5	1200 ± 184	13.5 ± 5.5	15 ± 5	0.63 ± 0.03
WF50(155)	18.5 ± 2.5	1450 ± 200	6 ± 4	8 ± 4	0.52 ± 0.02
CF10(155)	40 ± 8	1000 ± 103	29 ± 4	30 ± 4	0.68 ± 0.09
CF30(155)	27.5 ± 7.5	1010 ± 110	18.5 ± 6	19.5 ± 65	0.63 ± 0.04
CF50(155)	24 ± 6	1070 ± 160	18.5 ± 6.5	19.5 ± 6	0.53 ± 0.02
		Hot compaction at 165°C			
cPP/PP(165)	24 ± 1	966 ± 168	6 ± 0.5	20 ± 3	0.79 ± 0.05
WF10(165)	36 ± 10	950 ± 109	21 ± 4	22 ± 4	0.77 ± 0.04
WF30(165)	18.5 ± 2.5	1185 ± 70	7 ± 5	8 ± 5	0.77 ± 0.03
WF50(165)	15 ± 0,5	1462 ± 220	3 ± 0.8	3.5 ± 1	0.64 ± 0.21
CF10(165)	22.5 ± 4	1020 ± 88	9 ± 8	12 ± 7	0.75 ± 0.03
CF30(165)	16 ± 0,5	1160 ± 100	4 ± 0.5	7 ± 2	0.77 ± 0.04
CF50(165)	14 ± 1,5	1270 ± 108	3.5 ± 2.5	4 ± 3	0.62 ± 0.02
		Hot compaction at 175°C			
cPP/PP(175)	24.5 ± 1.5	809 ± 65	9 ± 1.5	312 ± 41	0.85 ± 0.02
WF10(175)	21 ± 0,5	910 ± 120	6.5 ± 0.5	8 ± 1.5	0.81 ± 0.04
WF30(175)	18,5 ± 0.5	1255 ± 95	4 ± 0.5	5 ± 1	0.86 ± 0.02
WF50(175)	14,5 ± 1	1600 ± 160	2.5 ± 1	3 ± 1	0.59 ± 0.18
CF10(175)	20.5 ± 1	1030 ± 150	5 ± 0.5	16 ± 3	0.78 ± 0.01
CF30(175)	16.5 ± 0.5	1020 ± 95	5 ± 0.5	8 ± 2	0.76 ± 0.01
CF50(175)	13 ± 0.5	1200 ± 85	2 ± 0.5	2.5 ± 0.5	0.72 ± 0.02

The received summary of impact-strength measurements [Figure 7(c,f)] shows a big influence of processing temperature on results. In connection with samples filled with 50 wt % wood floor was observed a significant decrease of impact strength for all series of measurements. In the context of the differences associated with temperature of compression molding, the best possible properties were obtained for the samples prepared at 175°C while the worst for samples compressed at 155°C. In the case of samples filled with cellulose flour the decrease of impact strength was observed. It can be certainly stated that impact strength is more dependent on the concentration of the natural filler, but increasing temperature of process has a beneficial influence on the structure of composites subjected to dynamic loads.

Materials Morphology

The SEM images of obtained hybrid polymer composites were presented to show the biggest differences in the morphological properties of the samples. In connection with reference materials obtained from pure bicomponent fibers [Figure 8(a)] we can see visible differences of structure relate to the presence of polypropylene fibers in the compacted material. As was shown, the SEM micrographs for the samples cPP/PP(155°C) the number of remaining fibers in the materials is surprisingly low. As expected, for the highest temperature of compression molding

process, samples cPP/PP(175°C), there was observed a homogenous structure of polymer blend. At the lowest temperature of processing was noted a very low amount of polypropylene fibers, which cause a lack of self-reinforcing effect.

Based on SEM micrograph [Figure 8(b)] for sample WF10(155°C) it was confirmed a big amount of remaining polypropylene fibers in the material structure. Because of their significant contribution in the composite in comparison with the amount of wood, they are the major factor caused the increased strength of obtained samples. Regarding discussed samples, rapid increase of polypropylene fibers amount was brought by the addition of wood. The particles of wood in the compacted composites cause the reduction of the stress relaxation during melting of the outer layer of bicomponents fibers. Finally, after bonding the greater amount of oriented structures was remained in the product.

For both structures showed in Figure 8(c) the main role in forming the mechanical properties of the composites plays the wood filler. What is interesting, despite of the predominant amount of wood particles within structure of composites, on the SEM images of sample WF50(155°C) the unmelted polypropylene fibers were observed. Their round section distinguishes from irregular wood particles. The presence of the polypropylene fibers was proved by other experimental results. This time large
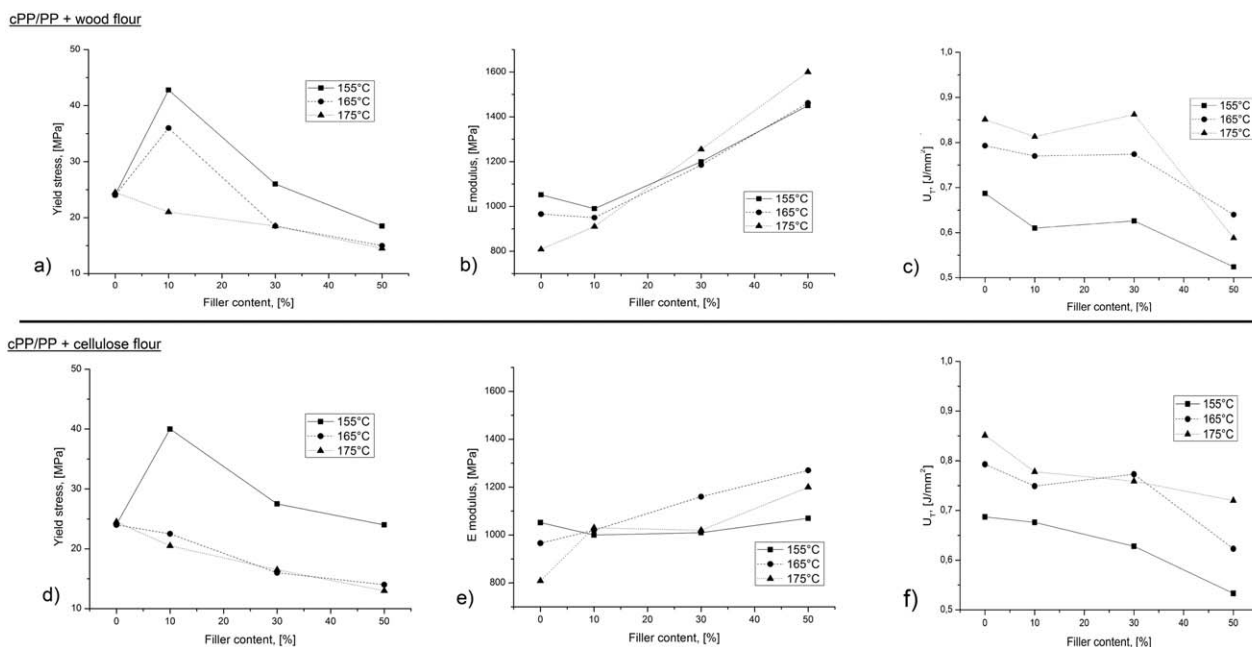
Figure 7. The mechanical properties presented in a form of yield stress, E modulus and impact strength changes, for wood flour based composites (a–c) and cellulose based composites (d–f).

percentage of polypropylene fibers in the structure has a positive, but not dominant influence on reinforcement of product.

The change of polymer filler on cellulose particles is shown in the form of clear change in the filler morphology [Figure 8(d,e)], compared to the wood particles. In the event of sample CF10(175°C), the fracture surfaces showed small but regular shape of the filler particles. The structure of the polymer matrix is homogenous, because all the fibers were melted. In the case of lower processing temperature for CF10(155°C) samples a large percentage of the composite structure consist of polypropylene fibers, which is a reason of enhancement strength of hybrid composites.

The increase of cellulose content in the polymer composites cause evident changes in the structure. For samples presented in Figure 8(e), lower temperatures during compression molding
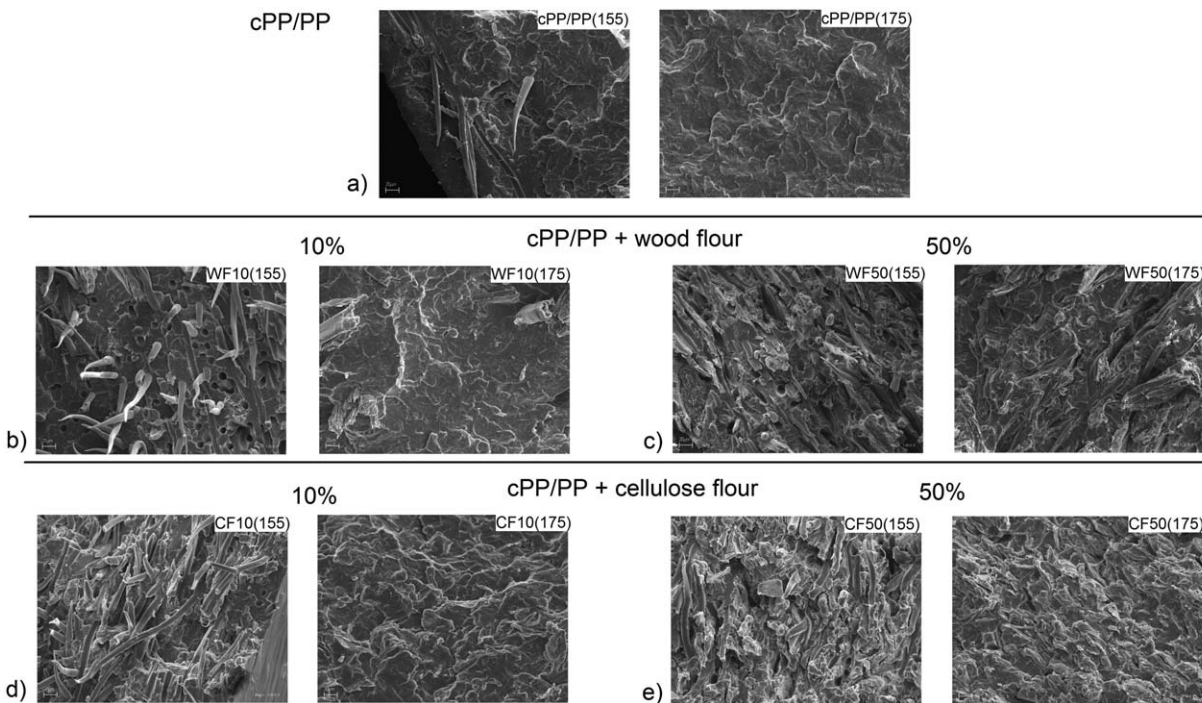

Figure 8. The structure of prepared composites for materials obtained from cPP/PP fibers (a), filled with wood flour (b,c) and cellulose flour (d,e). Each pair of SEM images show samples prepared at 155°C (left) and 175°C (right).

for CF50(155°C) composites cause the presence of unmelted polypropylene fibers. The increase of temperature processing, for CF50(175°C) samples induces a complete melting process of oriented polypropylene structure.

CONCLUSIONS

In this work, the fabrication of hybrid composites based on polypropylene and natural fillers (wood flour or cellulose flour) was carried out, the used compression molding method allowed to obtain the expected composites structures. The low temperature of compression molding preserves the polypropylene fibers from melting, while the addition of natural fillers holds the fibers in a tensioned state. The consequence of this two factors is the enhancement strength of hybrid system, which can be confirmed by yield stress of obtained self-reinforced structures. The other mechanical characteristics are dependent on the content of filler. For example, impact strength test for hybrid composites showed the positive influence of increasing temperature on the structure of composites subjected to dynamic loads. The increasing amount of filler, by the contrast induces the drop of impact strength. Additionally, the tensile properties (E modulus) increase with the increasing filler content (wood flour as well as cellulose flour). For highest filler content, the drop on yield stress was observed. The processing method develop during the research meet the expectations related to the possible further applications. The obtained hybrid composites are competitive to standard WPC materials in terms of processing and properties.

ACKNOWLEDGMENTS

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